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Norma-Hoffman Constant Temperature Bath Shown With Grease Stability Bomb and New Evaporation Test Cell.
(Page 5)

PUBLISHED BY THE
NATIONAL LUBRICATING GREASE INSTITUTE

In This Issue . . .

"PRECISION" Norma-Hoffmann BATHS AND BOMBS

For testing Chemical stability of Lubricating Grease

Lubricating greases in machine parts unused for long periods occasionally undergo oil separation, turn rancid and harden sufficiently to prevent free movement of rotating parts. Some lubricating greases are more stable than others, therefore, the selection of greases that have good storage stability is important in specifying lubricants for gears and transmissions, electric motor bearings, ball and roller bearings, sleeve bearings and similar applications.

The Norma-Hoffmann Bearings Corporation sponsored researches at the Pennsylvania State College and conducted extensive tests in its own research department, resulting in the development of an accelerated test which predicts the storage life of soda-base lubricating greases.

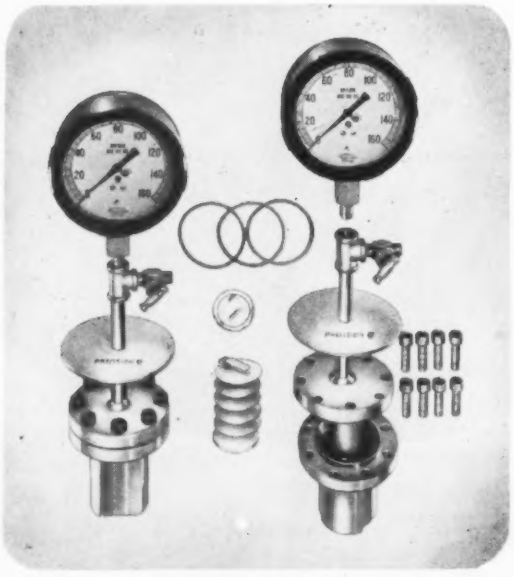
It was found that oxygen absorption data could be obtained conveniently on small samples with a sensitivity not attainable by methods involving usual chemical analysis. The apparatus consists essentially of a steel bomb in a constant temperature bath. Each bomb is fitted with a suitable valve for flushing and filling with oxygen; also a pressure gauge to indicate drops in pressure resulting from the absorption of oxygen by the sample of grease.

This test makes it possible to specify definite storage stability characteristics for lubricating greases, either in terms of the number of hours for the induction period or the rate of oxygen absorption in c.c. per hr. during the induction period. The method permits a reasonably rapid check on shipments for uniformity between batches, which is one of the important considerations in providing the best possible lubricant for anti-friction bearings. It also enables grease makers to control and improve their raw materials aand processes so as to produce a more stable and uniform finished product.

REFERENCES

Product Engineering, June 1936—"Determining Quickly the Storage Stability of Lubricating Greases," by F. L. Wright, Metallurgist, and W. A. Lutz, Chemical Engineer Research Laboratories, Norma-Hoffmann Bearings Corporation.

A.S.T.M. Preprint No. 94, 1938—"Some Applications of an Accelerated Test for Determining the Chemical Stability of Lubricating Greases," by F. L. Wright, Metallurgist, and H. A. Mills, Research Engineer Research Laboratories, Norma-Hoffmann Bearings Corporation.



FEATURES

The Norma-Hoffmann Constant Temperature Bath is supplied with removable metal rack for 3 bombs. Interior of tin-lined copper, exterior of galvannealed iron coated with metallic aluminum spray; heavily insulated to insure temperature uniformity. A motor-driven propeller stirrer agitates the heating medium for uniform circulation around bombs; patented Low-drift immersion heating units of low wattage per unit length, insure instant thermal response. Operation of the control heater is signalled by a Neon pilot light and governed by a sensitive mercury thermo-regulator working in conjunction with a super-sensitive relay concealed inside the bath housing. Overall dimensions 18" dia.; 21½" in height. **Write for detailed Bulletin No. 3410-Y.**

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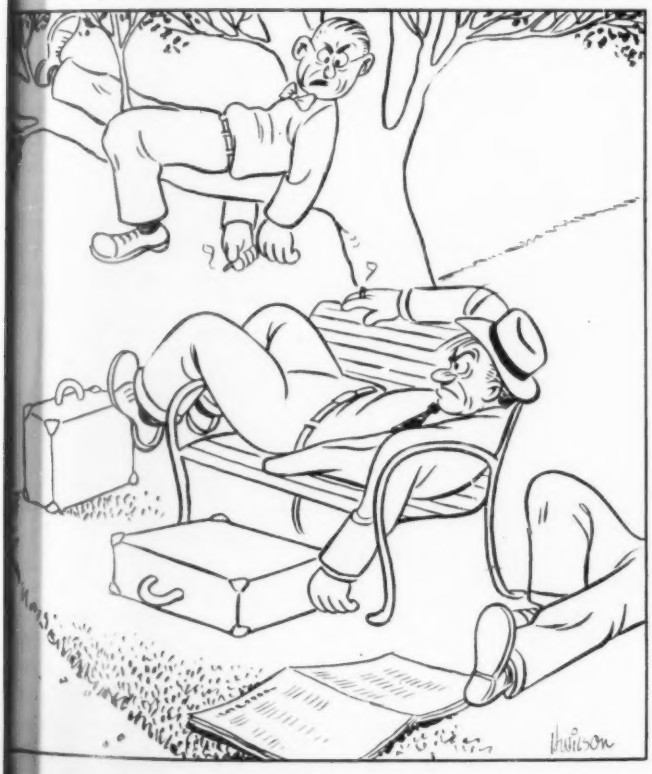
...L. G. I. ANNUAL MEETING

...ent of the Year to Be Staged at Edgewater Beach Hotel on the Shores of Lake Michigan in the Windy City

...tes of Activity Set as October 11, 12 and 13; Make Your Reservations Now

October 11-13, 1948, will see the N. L. G. I. increasingly popular Annual Meeting take place at the popular Edgewater Beach Hotel. The record attendance now fully indicated seems to justify early reservations for everyone. All reservations can be made through: Mr. H. J. Lacenere, Director of Sales, 5300 Sheridan Road, Chicago, Illinois. If you plan to join the crowd and attend, then make your reservations now and assure yourself of a place to sleep. Roosting in trees and park benches may be duck soup for Tarzan, but you will find it somewhat on the rough side.

"I made my hotel reservation in June for the Annual Meeting."

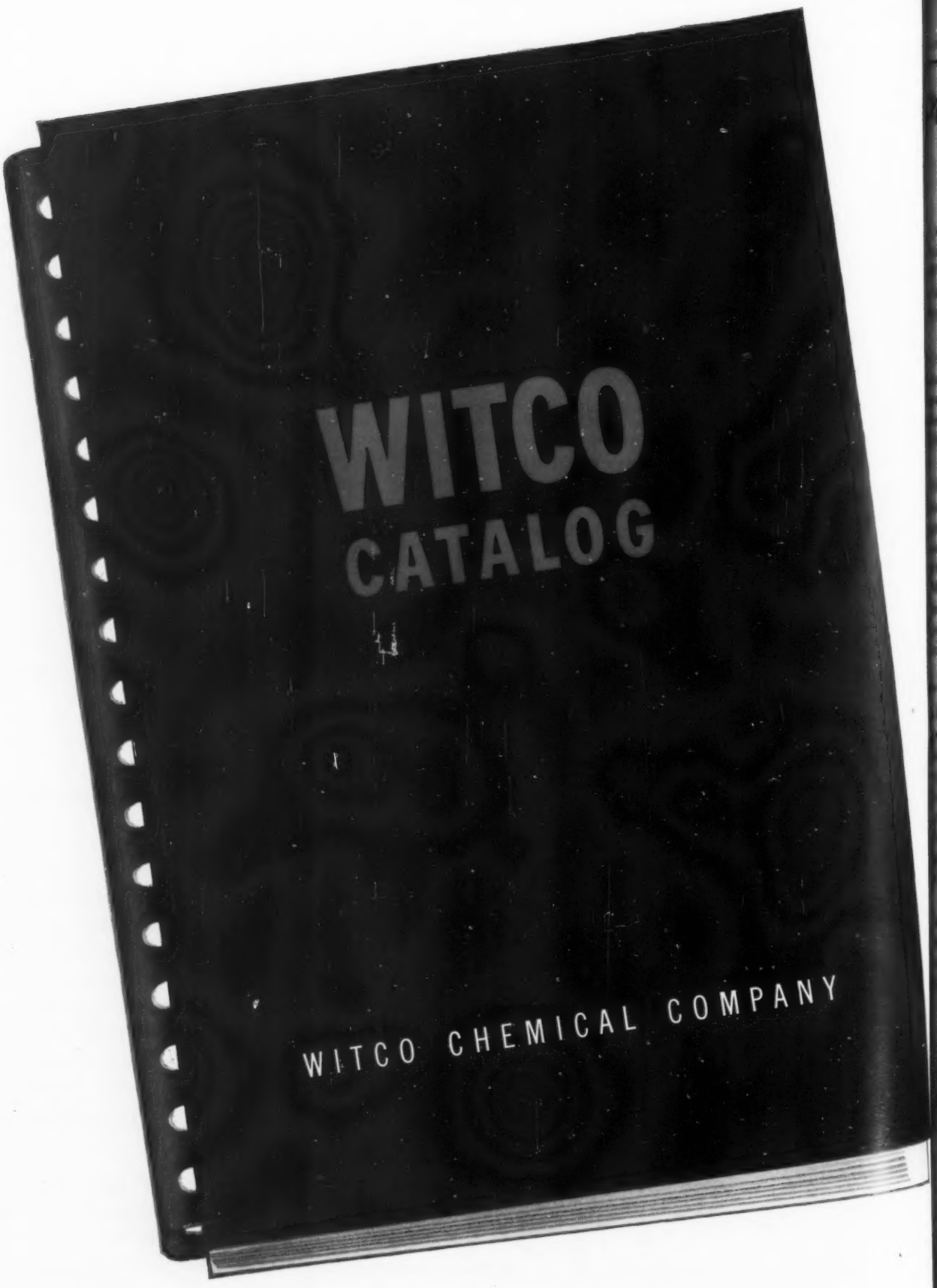


"We made our hotel reservations the day before the Annual Meeting."

YOU CAN'T LOSE

One of the most valuable, if not the most valuable, gain you can make is the benefit from personal contacts made during your three day stay. There is no other way you can informally discuss problems of mutual interest with others in your industry. You can't visit the headquarters of all the varied industries represented, but you can meet the executive officers of these organizations, exchange experiences and first hand information on the solution of your common problems. Old friendships are renewed and strengthened, and the new ones you will make can only be done in person.

Under the able leadership of Program Chairman, B. G. Symon, a completely stimulating group of speakers and subjects have already been assembled for your benefit. You will again reap the benefit of hearing and discussing the latest developments in your industry. The advantages gained by attendance can only be obtained through your presence there, a proxy is no good on this deal.



Send for your copy of the completely revised edition just published . . .

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New Members-Welcome

GEUDER, PAESCHKE & FREY CO.

324 North Fifteenth Street
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Since 1880, Geuder, Paeschke and Frey Co. (or G. P. & F., as many of our friends call us) has grown considerably. When grandmother was a girl, we thought we were doing all right in our little "Tin Shop."

Little did our founders realize that from this small beginning we would one day become a multi-million dollar company, occupying over 577,000 square feet of floor space—manufacturing thousands of different items.

Originally G. P. & F. Co. specialized in tin and black iron ware. This spread to facilities for enameling, tinning and galvanizing. Their reputation as experts in this field soon spread and lead to the establishment of a thriving Contract Manufacturing Division. This division serves other industries such as automobile, refrigerator, and washing machine manufacturers.

Acquiring the patent right to the "Wedg-Seal" steel shipping pail lead to the addition of a modern steel package plant. This division serves the grease, oil, chemical and paint industries. Of special interest to the grease and oil industry is their line of grease pails, oil drums, flour pails and kerosene type oil cans. The latest addition to this division is the 25 pound and 35 pound E-Z-FILL Grease Gun Loader pail.

The grandsons of the founders, who have taken over the leadership of G. P. & F., have pledged themselves to continued progress to the industries they are now serving.

ABOUT THE COVER . . .

Norma-Hoffman Constant Temperature Bath Shown With Grease Stability Bomb and New Evaporation Test Cell

The Norma-Hoffman test is used extensively to determine the resistance of lubricating greases to oxidation when stored under static conditions for long periods of time. The sample of grease is oxidized in a bomb heated to 210° F. and filled with oxygen to a pressure of 110 p.s.i. The pressure is observed and recorded at stated intervals. The degree of oxidation after a given period of time is determined by the corresponding decrease in oxygen pressure. Results of tests make it possible to specify definite storage stability characteristics for lubricating greases, and to permit a reasonably rapid check on shipments for uniformity between batches.

The Norma-Hoffman constant temperature oil bath assures temperature uniformity. Tests are usually carried out at 210° F. 1°.

The new improved Evaporation Test Cell may also be conveniently used in the Norma-Hoffman bath simultaneously while a grease stability test is being carried out. The Evaporation Test Cell was designed for the determination of evaporation loss of lubricating greases or low-viscosity oils which are intended for low temperature service. The sample of lubricant in a closed evaporation cell is placed in the bath maintained at 210° F. Heated air is passed over its surface for 23 hours at a specified rate. The evaporation loss is calculated from the loss in weight of the sample. This test provides a means of characterizing lubricants with regard to their tendency to lose oil by evaporation while in service.

The INSTITUTE SPOKESMAN

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THE NATIONAL LUBRICATING GREASE INSTITUTE

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Kansas City 2, Mo.

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N.L.G.I. EXECUTIVE SECRETARY DID NOT WIN BICYCLE OR PAIR OF ROLLER SKATES

Word seems to have gone the rounds that Harry F. Bennetts won something or other for doing whatever it was. Sufficient curiosity has been evidenced as to what was won and why, that some explanation is in order.

Your Executive Secretary won the 1947 Award of Merit presented to him by the American Trade Association Executives. Presentation took place in Washington, D. C. on April 26, 1948, at a luncheon. The address of presentation was made by Assistant Secretary of Commerce David Bruce.

The Award is given annually by a Jury of Awards to those Associations which, in the opinion of the jury, have done an outstanding job in rendering distinguished service to industry, to industrial development at large, or the public as a whole or some important segment of the public. The presentation may cover one or more activities of the association during the period to be covered by the presentation.

The 1947 Jury of Awards consisted of: Chairman, Honorable W. Averill Harriman, Secretary of Commerce; Earl Bunting, Chairman of the Board, National Association of Manufacturers; Alfred Reeves, Advisory Vice President, Automobile Manufacturers Association; Earl O. Shreve, President, Chamber of Commerce of the United States; Homer B. Vanderblue, Dean, School of Commerce, Northwestern University.

Unfortunately this award will go to his previous affiliation: the Electric Association of Kansas City. This award "was won because of its aggressive and

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RAPID METHODS OF GREASE ANALYSIS

Report by Section I on Chemical and General Laboratory Tests for Lubricating Grease of Technical Committee G on Lubricating Grease, of Committee D-2 on Petroleum Products and Lubricants

Section I on Chemical and General Laboratory Tests for Lubricating Grease, of Technical Committee G on Lubricating Grease undertook a study of methods of grease analysis because of the generally recognized need for improvement in the accuracy and applicability of the present A.S.T.M. Standard Methods of Analysis of Grease (D 128-40).[2] During the course of this study, it became evident that a procedure accurate enough for referee work may be too time-consuming and complex for a rapid control test. Consideration was given to a study of rapid methods particularly adaptable to control purposes, even though it was realized such procedures might not be sufficiently accurate for referee work.

Particular thanks are due to the following members of Section I, who in response to a survey, kindly submitted for consideration five procedures which they had found useful in their work on lubricating greases:

C. J. Boner and G. A. Williams, Battenfeld Grease & Oil Corp.
W. C. Bryant, Swan-Finch Oil Corp.
H. A. McConville, General Electric Co.
N. J. Gothard, Sinclair Refining Co.
W. S. Palmer, The Texas Company

Each method is described briefly in the following paragraphs, although the complete procedures are appended for more detailed reference. A summary of the comments on each method made by members of Section I is included also under the brief description of each method. In this connection it should be pointed out that these comments, except for condensation, are largely unedited; hence the points mentioned may not be regarded by all as being of equal weight.

Determination of Oil in Lubricating Greases (Boner and Williams)

Thtis is a qualitative method of separating mineral oil from lubricating greases, principally for the purpose of obtaining tests on the mineral oil component. Calcium soap greases are heated in the presence of 1 per cent hydrated lime. Sodium soap greases are mixed with approximately five parts of water and boiled. Aluminum soap greases are converted to soda soap by heating with caustic soda and subsequently treating with boiling water.

Although this method provides a rapid means of segregating a quantity of mineral oil, the characteristics of the separated oil may be influenced by the following factors:

(*a*) Adsorptive fillers may preferentially absorb components of the mineral oil,

(*b*) Calcium soap may not be completely removed,

(*c*) Boiling water hydrolyzes sodium soap, but this may be overcome by the substitution of 50 per cent alcohol, and

(*d*) Some sodium greases give serious emulsification difficulties when boiled with water.

Swan-Finch Control Method (Bryant)

The soap content is determined by calculation after decomposing the grease with crystalline potassium bisulfate, extracting with petroleum ether, and titrating the extract with 0.2 *N* KOH. A method for recovery of the mineral oil involves extraction of the mineral oil from the titrated solutions with petroleum ether and then evaporating the petroleum ether. Fillers may be determined prior to titration by filtration.

Although this procedure is an improvement over Method II of Methods D 128, the use of hydrochloric acid may be preferable for light-colored greases. The results may be subject to some error because it is questionable whether all the neutral fat is removed or whether the soap is completely removed from the ether solution. Some error may be introduced because there is no correction for free fatty acid, and petroleum ether insoluble materials such as asphaltenes are determined as fillers. Difficulties may be experienced with emulsions and also with end points of dark solutions.

General Electric Method for Soap (McConville)

This method involves decomposing the grease by shaking with HCl and petroleum ether, washing the mixture free of mineral acidity and titrating the oil layer with 0.5 *N* alcoholic KOH.

This method appears to be a slight modification of Section 12 (*b*) in Method D 128. The use of heat in decomposing the grease would save time. The method provides no correction for free fatty acids and is difficult to apply to dark oils.

Sinclair Method for Grease (Gothard)

This method is applicable only to sodium soap greases containing no free fat, particularly to railroad types of greases of relatively high soap content. The mineral oil is determined by extracting with 86-deg. naphtha and weighing the residue after the solvent has been evaporated. Free alkali is determined by adding excess *N* HCl and back titrating with caustic solution. The total alkali is determined by the Methods D 128-37 for sulfated ash. Combined alkali is obtained by subtracting the free alkali from the total and the soap is calculated from the combined alkali.

NOTE—DISCUSSION OF THIS PAPER IS INVITED, either for publication or for the attention of the author. Address all communications to A.S.T.M. Headquarters, 1916 Race St., Philadelphia 3, Pa.

[1] Report presented at the January, 1947, meeting of Technical Committee G in Washington, D. C.

[2] 1946 Book of A.S.T.M. Standards, Part III-A, p. 201.

The substitution of 95 per cent alcohol would prevent hydrolysis of the soap. The total alkali will be high if neutral sodium salts are present.

Texas Method for Grease (Palmer)

This method is applicable only to sodium soap greases of the railroad type of relatively high soap content. The sample is ignited in a platinum or porcelain crucible and heated to a dull red heat. The crucible and contents are boiled for 2 hours in distilled water, and after cooling the water solution is titrated with 0.25 *N* hydrochloric or sulfuric acid using a methyl orange indicator. Soap is calculated from the difference between total alkali as determined above and free alkali.

The method is extremely limited in scope and, like other rapid methods, presupposes knowledge of the composition and manufacture of the grease.

Although written comments were limited in number, the discussion at the June 24, 1946, meeting of Technical Committee G held in Buffalo, can be taken as representative of the feeling of the entire membership. The following quotation taken from the minutes of that meeting summarizes this feeling:

"The over-all conclusion was that such methods were inapplicable for referee work since they were definitely predicated on a presupposed knowledge of the composition of the greases. This was em-

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RAPID METHODS OF GREASE ANALYSIS

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phasized, pointing out that such knowledge of manufacture and composition was, of course, not available to the consumer. Therefore, these short-cut methods could be used only with extreme caution and with a full realization of their limitations."

In other words, the rapid methods could be used by a manufacturer as a matter of routine plant control or could be used for a rapid means of routine inspection by a purchaser, provided there was a full understanding between the seller and the purchaser regarding the applicability of the rapid method and a realization that the A.S.T.M. method or any future revisions should be used for referee purposes whenever a result by the rapid method is questionable.

APPENDIX

Rapid Methods for the Determination of Oil in Lubricating Greases

By C. J. Boner[1] and G. A. Williams[1]

The modern laboratory is confronted with many of the same problems as other departments of industry. One of these is to secure results as promptly as possible. As a result of demands for quick results, methods have been devised for the separation of oils from lubricating greases which consume less time and are more simple than the standard A.S.T.M. method (Sections 15 to 20, (Methods D 128-37) A.S.T.M. Standards on Petroleum Products). If care is used in separating oils by these rapid methods, an oil of the same purity and characteristics can be obtained as by use of the standard method. Details of the methods proposed and comparison of oils extracted by the rapid method with the same oils secured by the standard method follow.

The general plan used for the quick separation of oil from calcium greases depends on the fact that most calcium-base greases, if completely dehydrated, will exhibit syneresis. In order to have an abundance of oil separate, it is best to use about 750 g. of greases below 250 penetration and 500 to 600 g. of greases above 250 penetration. The grease is weighed into a 3-qt. kitchen pan, either black iron or granite-ware, and about 1 per cent of hydrated lime added. It is then heated to 300 F. and held there with stirring until thinning is noted, which is usually about 1 min. The sample is then set off the fire to cool. It is then found that at 300 F. the water has left the grease and foaming has ceased. Oils will seldom be encountered in lubricating greases with flash points below 325 F. Hence the oil should not be changed by this treatment. If a very speedy determination is desired on the oil characteristics of the grease it is preferable to cool in a refrigerator. In any event, when a temperature of about 175 F. is reached, there will result a plastic mass. By working this mass with a spoon or spatula, oil separation will be hastened. Better still, leave the mass until it comes to room temperature when granular soap will separate leaving clear oil which can be poured off. It is preferable to strain the oil through a cloth filter.

[1] Battenfeld Grease and Oil Corp., Kansas City, Mo.

...se weave of cotton sacking material ...s been found to make the best filter, ...ving a speedy filtration yet delivering a ...ear oil. By the first procedure outlined ...ove—that is, working at about 175 F. ...almost twice as much oil is recovered ...is true if the mass sets undisturbed ...until cool. However, the oil in the first ...e is liable to contain flocks of soap. ...case these are evident, reheating to ...00 F. and cooling again will cause the ...oap to coagulate and settle. To assure a final oil free from soap and lime, the oil ...centrifuged before any characteristics ...are determined, using 100-ml tubes. In ...most instances, no sediment is obtained ...n centrifuging.

It may be found that some variation of this general plan will give best results for others. The added lime no doubt has two or three functions and this lime addition can be varied as to amount in most greases. It is believed that the added lime will saponify any free fatty acids and part, if not all, of the free fat which may be in the grease. Further, this deficiency in free fat or fatty acids will cause greater syneresis than otherwise. By using several times as much added lime as is recommended, a cloud in the oil generally results. The same condition has been found when oils alone are heated with hydrated

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RAPID METHODS OF GREASE ANALYSIS

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lime to 300 F. and then cooled. The addition of 0.1 to 0.25 per cent of filter cel, both to the grease and to the oil before filtering, has been tried as an additional aid in settling the soap. This seems to offer promise.

As to the limitations and merits of this method, it provides a quick method for the separation of oils from lubricating greases, particularly those containing light-colored and low-viscosity oils. As will be noted from Tables I and II, oils separated by this method check closely in viscosity with those separated by the standard A.S.T.M. method. In most cases, the rapid method recovers an oil somewhat lighter in color than the standard method. The darkening by the latter method may be due to the action of hydrochloric acid or more probably to heating to drive off solvent. Of course, in this more rapid method there is a saving due to no use of solvents but the fire hazard due to their use is removed. Little glass apparatus is used with a consequent saving in breakage. Where fillers such as graphite are present, they are carried down by the mass of soap.

No doubt most laboratories have had customer complaints to the effect that the mineral oil in a grease shipment is not according to specifications. In some such cases, even though the standard A.S.T.M. method was followed, the following errors were found: incomplete soap decomposition by acid with consequent high viscosity report on oil; solvent left in recovered oil resulting in report of low viscosity; oil darker than specifications which was traced to excessive heating to drive off solvent.

This rapid method and the one following were devised to prevent the above discrepancies, if possible. At the same time, the saving in time is the biggest argument in its favor. It is doubted whether most laboratories can complete a separation of lubricating grease by the standard A.S.T.M. method in less than 5 to 6 hrs. With this rapid method they should be able to recover and test the oil in about one-fourth this time.

Naturally, this is not a quantitative method for mineral oil in lubricating greases. Further, it is not applicable to rosin greases. It will apply to greases made either from fat acids or from whole fat. No work has been done with greases with added materials such as extreme pressure bases. In the case of calcium greases of high soap content, such as No. 5 Cup, it is sometimes difficult to obtain oil separation by the rapid method. In such instances a small amount of solvent will leach out oil which is afterwards reheated and filtered.

Of course, the same procedure is applicable to smaller quantities of grease where the final viscosity determinations are made by the suspended-level or modified Ostwald viscosimeter.

Those not familiar with this method will no doubt wonder what the effect of small quantities of impurities may be on

the recovered mineral oil. Therefore, impurities, such as might be expected, were added to three representative mineral oils and comparative viscosities determined. In each instance, the added material was heated with the oil to 300 F., cooled to room temperature, and centrifuged before viscosity determinations were made. The results are given in Table I.

It is felt that the additions of Table I are much above the possible percentage likely to be encountered in oil recovered by the proposed method. Hence, it is reasonable to conclude that close checks may be expected by this method with the oil actually entering the particular grease.

TABLE I.—SAYBOLT VISCOSITIES ON STRAIGHT AND COMPOUNDED MINERAL OILS.

Oil or Compound	Saybolt Universal Viscosities, sec.
300 Vis. at 100 deg. oil	325 at 100 F., 142 at 130 F.
300 Vis. + 2 per cent 42 titer fat	317 at 100 F., 139 at 130 F.
300 Vis. + 2 per cent 38 titer fat acids	310 at 100 F., 138.5 at 130 F.
300 Vis. + 1 per cent lime	327.5 at 100 F., 141.5 at 130 F.
300 Vis. + ¼ per cent NaOH and 1 per cent water	324 at 100 F., 141 at 130 F.
2000 Texas oil	87 at 210 F.
2000 + 2 per cent 42 titer fat	84.5 at 210 F.
2000 + 2 per cent 38 titer fat acids	83.5 at 210 F.
2000 + 1 per cent lime	87 at 210 F.
2000 + ¼ per cent NaOH and 1 per cent water	86.5 at 210 F.
160 bright stock	165 at 210 F.
160 + 1 per cent 42 titer fat	163 at 210 F.
160 + 1 per cent 38 titer fat acids	162 at 210 F.
160 + 1 per cent lime	165.5 at 210 F.
160 + ¼ per cent NaOH and 1 per cent water	164.5 at 210 F.

This is borne out by Table II showing representative results obtained by the rapid method and by the standard method on the same grease samples.

For the rapid separation of mineral oil from soda-base grease, we depend on the solubility of the soap in water. By carry-

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RAPID METHODS OF GREASE ANALYSIS

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ing out a boiling process in the proper manner it is surprising how rapidly and accurately results can be obtained on the oil component of a grease.

In detail—about 200 to 300 g. of the lubricating grease is placed in a 3- to 4-qt. flat pan together with 1200 to 1500 ml. of boiling water. Boiling is continued until the grease is decomposed and a clear layer of oil appears on the surface. The water is then siphoned off and boiling repeated with fresh water. After 10 to 15 min. this water is removed and fresh added. This procedure is repeated until the wash water on cooling remains clear. Normally four boilings will free an oil of low viscosity from soap, while a heavy oil will require twice this number. It is preferable to make the final separation in a separatory funnel and follow by centrifuging. If the oil is not perfectly clear it should be heated above 212 F. until dry. During the last boiling the addition of 5 ml. of acetic acid will decompose any soap remaining. Further, the addition of a small amount of alcohol to next to the last wash may assist in obtaining a clear oil.

The success of this method depends largely on very intimate contact of the oil with the water. By using a wide pan so that a thin layer of oil is present, this condition prevails. If this method is used it will be found convenient and time-saving to have an abundant supply of boiling water on hand.

As with calcium greases, this method is not quantitative. Also, neither method provides for separation of oils from mixed calcium and soda greases. Of course, the most satisfactory results are obtained with low viscosity oils, but heavy oils do not offer any more trouble than by the standard method and possibly less.

That satisfactory results can be obtained on oils separated by this rapid method is shown in Table III.

For the rapid separation of oil from aluminum-base greases, the aluminum soap is converted to soda soap and the dissolved as is done with regular soda greases. About 200 to 300 g. of aluminum grease is heated with 20 to 25 per cent of its weight of 35 to 40 Baumé caustic soda solution. Heating should

TABLE II.—SAYBOLT UNIVERSAL VISCOSITIES OF OILS SEPARATED FROM CALCIUM LUBRICATING GREASES

Type of Grease	A.S.T.M. Method, sec.	Rapid Method, sec.	Ash, per cent
Air drill	473 at 100 F.	471 at 100 F.	0.01
Cylinder stock chassis	160 at 210 F.	161.5 at 210 F.	
		161 at 210 F. (0.2 per cent filter cel used)	
Chassis lubricant	583 at 100 F.	588 at 100 F.	0.02
		586 at 100 F. (0.02 per cent filter cel)	
F. A. gun	301 at 100 F.	303 at 100 F.	0.01
Graphite Cup	299 at 100 F.	300 at 100 F.	0.004
No. 3 Cup	328 at 100 F.	334 at 100 F.	
Gun	303 at 100 F.	305 at 100 F.	
No. 4 Cup	91 at 100 F.	87.5 at 100 F.	
		87 at 100 F. (no extra lime added)	
No. 4 Cup	140 at 130 F.	137 at 130 F.	0.06
No. 3 Cup	296 at 100 F.	308 at 100 F.	
Dark gun	90 at 210 F.	93 at 210 F.	
Crank pin cup	301 at 100 F.	314 at 100 F.	0.07
Cylinder stock chassis	81 at 210 F.	84 at 210 F.	0.09

TABLE III.—SAYBOLT UNIVERSAL VISCOSITIES OF OILS SEPARATED FROM SODA-BASE GREASES

Type of Grease	A.S.T.M. Method, sec.	Rapid Method, sec.	Ash, per cent
No. 2 Sponge	140.5 at 130 F.	140 at 130 F.	
Wheel bearing	369 at 100 F.	370 at 100 F.	
Wheel bearing	75 at 210 F.	82 at 210 F.	0.09
Universal joint	92 at 210 F.	95 at 210 F.	
No. 3 Sponge	340 at 100 F.	347 at 100 F.	0.036
No. 3 Sponge	307 at 100 F.	310 at 100 F.	

in an Erlenmeyer flask to prevent danger of spattering. It is essential that the caustic solution be mixed very thoroughly with the grease during heating which should consume at least three-quarters of an hour. Following this heating, the flask is filled with water when a cake of oil and soap will result in a body of caustic water. The latter can be poured off. If very quick results are desired, boiling with water can be carried out at once. If not, by the following morning considerable clear oil will have separated from the cake in most instances. In any event, the oil must be freed from soap by boiling with water as is done in the case of soda-base greases before it is used for testing.

While this method is successful with strong caustic solution, it is not dependable with weaker solutions. Apparently sodium aluminate is formed and, of course, the fat acids set free are converted to soda soap. While this involves two steps, it is found that the method consumes less time than the regular A.S.T.M. method where characteristics of an oil in an aluminum grease are desired. That the method compares favorably with the standard method is shown by the results in Table IV.

TABLE IV.—SAYBOLT UNIVERSAL VISCOSITIES OF OILS SEPARATED FROM ALUMINUM-BASE GREASES

Type of Grease	A.S.T.M. Method sec.	Rapid Method, sec.
Gun	274 at 100 F.	262 at 100 F.
Rocker arm	183 at 210 F.	189 at 210 F.
Chassis	111 at 210 F.	115 at 210 F.
Chassis	128 at 210 F.	131 at 210 F.
Chassis	94 at 210 F.	99 at 210 F.
Gun	298 at 100 F.	299 at 100 F.
Gun	287 at 100 F.	284 at 100 F.

Oil in greases thickened by fillers only, as asbestos tractor lubricants, can be obtained either by heating and filtering or warming and pressing through cloth. Such lubricants, containing less than 10 per cent of filler, if heated to 300 F. will

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CHAIRMAN T. G. ROEHNER, DIRECTOR, SOCCNY-VACUUM LABORATORIES

- **"DELIVERY CHARACTERISTICS OF DISPENSING EQUIPMENT FOR LUBRICATING GREASES."**
- **SPECIFICATIONS FOR LIME FOR USE IN THE MANUFACTURE OF GREASES.**
- **K VALUES IN HEAT TRANSFER UNITS.**

In the Technical Committee Column in the March issue, mention was made of the panel organized to work on the Committee project entitled "Delivery Characteristics of Dispensing Equipment for Lubricating Greases." The first meeting of this panel was held on June 2nd and a summary of the minutes will be given in next month's Technical Column.

Relative to new activities, a survey is now under way to obtain the basis for a recommendation by the Committee to A.S.T.M. Committee C-7, Subcommittee III, regarding specifications for lime for use in the manufacture of greases. A.S.T.M. has suggested for consideration the following tests and limits:

Minimum % available calcium oxide	72%
Maximum % calcium carbonate	3%
Maximum % magnesia oxide	2%
Maximum % silica	1%

Mr. R. W. McAllister, of Arthur D. Little, Inc., is Chairman of the aforementioned Subcommittee III. His viewpoint is reflected in the statement that they "wish to adopt a specification which would be usable and used." The outcome of the survey of the interested Technical Committee members will be forwarded to Mr. McAllister for use at the June 22nd meeting of his group.

We recently had occasion to review some comments made by Mr. H. C. Zweifel, of the Richfield Oil Corporation, regarding problems which could be profitably undertaken by the Technical Committee. We are taking the liberty of quoting the following paragraph therefrom:

> **"A concern of all manufacturers is the efficiency of the plant. New mechanical units are used in every branch of the oil industry but relatively few have been developed for grease manufacture. If the necessary data were available it may be possible to obtain more satisfactory engineering assistance in design. For example, little is known regarding K values in heat transfer units; and few are willing to do more than guess regarding the equipment most**

...uitable for pumping of greases in ...quantity. The rate of mixing or the ...ntimacy of contact, between reacting ...gents in an oil medium, has not been ...tudied beyond the phase of practical ...pplication."

The above paragraph has been included in this Technical Column not only because it contains sound ideas, but also with the hope that it will stimulate other members to present other attractive ideas and suggestions as to how work thereon may be initiated by the Committee.

N. L. G. I. EXECUTIVE SECRETARY DID NOT WIN BICYCLE OR PAIR OF ROLLER SKATES *(Continued from page 5)*

successful trade promotion and public relations campaign. In its area, all types of business firms in the electric industry were induced to unite in personnel training programs, in store modernization, in market research, and in a sound plan of business ethics."

Adding his own statement to the above, the N. L. G. I. Executive Secretary devoutly hopes to be able to do even more for this organization.

President's Column

by J. R. CORBETT, President N.L.G.I.

OPPORTUNITIES FOR THE COLLEGE GRADUATE IN THE LUBRICATING GREASE INDUSTRY

Never have the prospects of a college graduate seemed brighter than they are today. Even though the world at large is in anything but a settled condition, the future looks promising. Business, on the whole, continues on the upgrade. For most graduates of colleges there are a multiplicity of outstanding opportunities.

But certainly there are no greater opportunities anywhere than in the field of lubrication.

This message is directed at those bright, young men who have just departed from the cloister of ivy-covered walls to find their rightful places in the world at large. We'd like for all of you young men to realize this: *Never before has the field of lubrication had so much to offer you.* And never before has the field of lubrication needed you as it does now.

This is truly the Machine Age. But we have merely scratched the surface. No one knows—in fact, no one dares even to dream—of the things that are ahead of us. And I don't mean that to sound ominous. Rather, I am thinking of the constructive side of the picture—the marvelous advancements that machinery will make, advancements that will lighten the load of man immeasurably, make him a freer a[nd] a happ[ier] individual.

And don't forget this, y[ou] graduat[es]. As machinery advances, so must lub[ri]cation advance. For mach[in]ery is de[-]pendent—*totally dependent*—on lubr[i]cation.

We have made giant str[i]des in the field of lubrication, but we have much to learn. There is much that we must do to the cause of machinery if we are to fulfill the hopes of the world.

We must employ all the resources at our command, and one of our great[est] resources is the well-spring of tal[ent] from our institutions of higher learning. For here lie the fine young minds—highly attuned and highly developed. Here is an abundance of new and construct[ive] ideas. Here lies invention. Here lies creative and executive skill. And here is ambition, the will to work and the will to succeed.

The field of lubrication covers a vast area of human endeavor. All types of men are needed. We need chemical engineers and mechanical engineers. We need research specialists. We need men who are well grounded in business administration—accountants, auditors, efficiency experts. We need salesmen

men who are sales minded yet possess, at the same time, a sound technical background. We need public relations men—men who can tell our story to the world in forceful, interesting language.

Yes, we need these men—all of them—and many more!

And never let a man think his labors will go unrewarded in our vineyard. As he contributes to the field of lubrication in work, energy, and initiative, he shall be rewarded amply—not only in monetary compensation, but—and this is a greater reward—in the satisfaction of playing a major part in the advancement of the most vital material force in the modern world.

RAPID METHODS OF GREASE ANALYSIS

(Continued from page 13)

filter through the same type of cloth used for calcium-soap separation. The oil can then be further centrifuged. With higher percentages of filler, warming to 125 F., followed by pressing through cloth, will give enough oil so that it can be further filtered and centrifuged.

This method, of course, does away with solvents and with prolonged heating of the oil which might change its characteristics.

Swan-Finch Control Method (Control Procedure 7-1000)

By W. C. Bryant[1]

SOAP CONTENT OF LUBRICATING GREASES, FILLERS[2] AND VISCOSITY OF MINERAL OIL

Purpose:

To determine the soap content as the proper base and to extract the mineral oil portion from the same sample for determination of the mineral oil viscosity.

Scope:

Proved satisfactory on soda base, lime base, and mixed soda lime base greases.

Equipment:

One 20-ml. Griffin low form pyrex beaker,
Two 100-ml. Griffin low form pyrex beakers,
One short stem 3-in. funnel,
Two 3-in. analytical funnels,
Three circles of Whatman No. 1 filter paper,
Two 500-ml. pyrex Erlenmeyer flasks,
One funnel support,
One hot plate or sand bath,
Two 500-ml. separatory funnels, and
One 300-ml. pyrex Soxhlet extraction flask, also kinematic viscosimeter and Gooch filtration[1] setups.

[1] Swan-Finch Oil Corp., Newark, N. J.
[2] See Paragraph (e) of this appendix.

Chemicals:

Potassium bisulfate c.p. crystals,
Petroleum ether c.p., pour point 35 to 60 C.,
0.2 *N* aqueous KOH solution,
Neutralized formula 30 alcohol or equivalent ethanol formulation,
1 per cent alcoholic phenolphthalein solution, and
Anhydrous sodium sulfate powder.

Method:

(*a*) In duplicate, weigh 10 g. of the sample to be analyzed, into the 100-ml. beakers. Care is to be exercised in placing sample in beaker so that none adheres to the side of beaker.

To each beaker add 10 g. of medium size crystalline potassium bisulfate.

Place samples on heat unit along with a 100-ml. beaker with 50 ml. of tap water. Adjust heat unit so that tap water is just at boiling point.

With moderate stirring, to give intimate mixture of bisulfate salt and sample, allow the sample to be heated until the soap has been fully broken. This is indicated by the cessation of bubble formation by the sample. The usual reaction time is: 20 min. for soda-base greases; and 40 min. for lime-base greases.

When reaction is complete, the beakers containing the samples are set on watch glasses and allowed to cool to room temperature, or slightly warmer.

(*b*) When the samples are cooled, prepare filter papers and funnels and set up Erlenmeyer flasks to catch the filtered petroleum ether extracts. Extract the samples with as little petroleum ether as possible but extract until the washings of petroleum ether are colorless, or until assured of complete extraction.

(*c*) To the extracts, add 50 ml. of the neutralized alcohol, and 5 drops of the phenolphthalein solution.

Titrate cold with the 0.2 *N* KOH solution until the phenolphthalein end point of the alcohol layer persists for 30 sec. Calculate the proper soap content from the titer factors and titrations.[3]

(*d*) The titrated extracts are combined in one of the separatory funnels and 20 ml. of KOH solution are added, the funnel shaken well. The alcohol layer is allowed to separate. The alcohol layer is drawn into the second separatory funnel which contains 50 ml. of the petroleum ether. The extract in the first funnel is washed with distilled water until the aqueous layer is neutral to phenolphthalein. Each washing is passed through the petroleum ether in the second separa-

[3] One ml. 0.2 N KOH = 0.06026 g. calcium oleate, log 2.780029
= 0.06086 g. sodium oleate, log 2.78433

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APID METHODS OF REASE ANALYSIS

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y funnel and discarded. When the tract is washed free of soap, as indicated neutrality of aqueous layer, the final sh water is drained off as completely possible. The extract is dried with 10 g. the anhydrous sodium sulfate powder the usual manner. The dried extract is ained through the short funnel, fitted ith filter circle, and collected in the xhlet flask. The petroleum ether is tilled off completely and the flask ith the oil residue is placed in a 105 C. ven for 10 minutes. Ten to 12 ml. of he hot, dried oil is decanted off into the 0-ml. beaker, and allowed to cool.

The cooled air is charged to the kinc- tic viscosimeter in the usual manner d the viscosity determined in the usual nner at the proper temperature.

(*e*) If fillers such as clay or graphite, tc., are present, filter the extracts from ction (*b*) through prepared Gooch lters, and the quantity of fillers can be determined in ordinary manner. The xtracts are collected in Erlenmeyer lasks, and the procedure followed as in sections (*c*) and (*d*).

General Electric Co. Soap Determination in Grease

By H. A. McConville[1]

The amount of sample taken depends on the approximate soap content of the grease. If it contains below 10 per cent of soap, take 10 g. of grease, and if it has 25 to 30 per cent soap, take 1 g.

The grease is weighed accurately into a 250-ml. beaker. Dissolve the grease as completely as possible in 75 ml. of petroleum ether, then pour the solution into a 250-ml. Erlenmeyer flask, washing

[1] Works Laboratory, General Electric Co., Schenectady, N. Y.

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BARIUM LUBRICATING GREASE

By C. J. Boner, Chief Research Chemist, Battenfeld Grease & Oil Corporation, Kansas City, Missouri
and G. W. Miller, President, Battenfeld Grease & Oil Corporation, North Tonawanda, New York

Introduction

Until the last few years the Lubricating Engineer who wishes to use a lubricating grease was limited in his choice to one which was water resistant, but low melting point; or one which was high melting point, but was not water resistant. Fortunately, there are now available Barium Base Lubricating Greases which combine water resistance and melting points up to 400° F. The experience with these lubricants in farm, automotive and industrial service has been very promising. This is particularly true of certain applications in steel mills. The purpose of this paper is to acquaint you with the characteristics and applications of Barium Lubricating Greases so that you may take advantage of this improvement in lubricants.

We hardly need to mention that lubricating greases are soap-thickened oils and that in order to prepare a satisfactory product, both the oil and the soap must be considered. It goes, without saying, that a well refined oil should be used. Over twenty years of study has led us to the conclusion that the proper soap is as much a lubricant as the oil and with this in mind we prefer a soap which maintains a body at high temperatures rather than fuses to a liquid. Properly manufactured barium soap has this quality as will be demonstrated later on a hot plate.

Before we tell you what applications Barium Grease may have in your plants and for your transportation equipment, it may be well to go over some of the reasons why you prefer to use lubricating greases rather than oils for some jobs. First, grease is preferable where atmospheric conditions are dirty. Under such conditions grease will seal the ends of bearings, thereby preventing dust and dirt from entering. We might digress here and point out the necessity of prevention of contamination of lubricants after the containers are opened. A good lubricating grease mixed with dirt is not as good as a mediocre grease which is clean. All reputable manufacturers of lubricating greases are extremely particular to see that products leave their plants with no contaminants in them. For example, Barium Lubricating Grease passes through Cuno Filters with a maximum opening of 0.008 of an inch before it is packaged. Second, lubricating grease is used when the location of bearings is such that they are inaccessible, since a grease lubricated bearing will require attention less frequently than one lubricated with oil. Third, lubricating grease is preferred where bearings operate under extremely severe conditions, such as high temperatures and extreme pressures. Fourth, bearings which must be lubricated in the presence of the washing action of water normally will render best service with lubricating grease.

Comparison With Other Lubricating Greases

You may be interested in just what happens to a lubricating grease in service. Grease structure consists of a soap matrix enmeshing oil. This matrix may be a mass of fibers, in some cases so small they cannot be distinguished even with magnification, or it may be a honeycomb structure. Irrespective of the form of this structure, it is what is responsible for the plasticity and resistance to flow of lubricating greases. When lubricating grease is subjected to shear and pressure as is the case when it is between two metal surfaces, the structure may change. The fibers may be broken and if this goes far enough, the product will soften and oil may be separated. The secret of a lubricating grease which will stand up in service is to have a structure which will not break down abnormally under the above conditions. This we have in Barium Lubricating Grease. One of the reasons for this can be seen if you compare the soap content of lubricating greases of the same consistency, but made with different bases. We have taken a consistency of 265 to 295 in each case. Barium base grease has approximately 25 lb. soap, an aluminum base grease 10.3% soap, a calcium base grease 11.5% soap, and a soda base grease from 8.5% to 14% of soap. Thus, Barium Lubricating Grease has almost twice as much soap as the other types. Another factor contributing to the stable structure of the subject grease is the heterogeneous nature of the fatty acids used in manufacturing the soap. We find it advantageous to have some very short chain length fatty acids together with some quite long chain length fatty acids to form our soap. The barium molecule can attach itself to two fatty acid molecules and we surmise that in our structure, we frequently have acids of different chain lengths attached to the same barium molecule. This provides a "complex" soap rather than simpler soaps which form the base of most lubricating greases.

This latter fact is also partly responsible for the more favorable reaction of Barium Lubricating Grease to temperature than is true of lubricating greases of other bases. It is a general rule that pure substances have sharp or abrupt melting points while mixed products vary from this rule. In the case of barium lubricating grease we have the latter condition and on a small hot plate we will demonstrate the difference in behavior to heat of Barium and those made from other bases. The temperature of the hot plate is adjustable from 100° F. and we will change the heat by 50° increments starting with 200° F. We will also incline the hot plate slightly so that as the grease starts to break down or the soap to melt it will be evident what happens. The first lubricating grease placed on the plate will be calcium base which melts and slides down as soon as it touches the hot metal. Next, aluminum base slides down almost as quickly. Now, two sodium base greases from different manufacturers: one fibrous, and the other of rather smooth texture. These do not move at 200° F. but one of them starts to melt at 250° F. and the other at 300° F. Following this, we will place a sample of lithium base grease on the hot plate. This type is being marketed with claims for high temperature qualities. If we wait long enough, this sample will move slightly on the plate at 300° F. However, we now place a sample of Barium Base Grease alongside it, and after a short interval raise the temperature to 350° F. You will then see that the lithium grease starts to melt and run off the plate. Since the Barium Base Grease still adheres to the plate, we will raise the temperature to 400° F. The subject grease still stays in place which indicates that in service if you employed this lubricant, it would remain in a bearing at or above this temperature when other types would run out.

Advantages of Barium Lubricating Grease

You will see that these tests demonstrate not only the high melting point of this type of lubricating grease, but also the quality of adhesion to metal, in

ich respect it also has superiority. This esion can be easily demonstrated if will spread a little barium lubricating se on a metal surface and then try wipe the surface clean. In the laboratory we cannot clean it from a surface ess we use solvents and even then one lication is not enough. This characristic, together with the tendency to el water is, no doubt, responsible for ability of this lubricant to protect tal surfaces against corrosion.

The high water resistance of barium ubricating greases is particularly imtant and has been confirmed by nuerous service tests. In the laboratory has been found that barium lubricating ease passes the navy water absorption d leaching tests of Specification 14L5. his test consists of circulating a ¼" ream of water at 150° F. over a quanty of lubricating grease held in a perated cone and determining the loss fter a 2-hour period. The qualities which ake Barium Lubricating Greases reistant to water are the fact that the soap es not hydrolyze and also that the soap oes not emulsify with water. As prouced, it is almost neutral, and even on rolonged boiling with water there is no ction on the soap. Sodium soap greases

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Malmstrom Appoints New Sales Representative

N. I. Malmstorm & Company of New York, America's largest processor of Wool Fat and Lanolin, announces the appointment of Mr. James L. Johnson to its sales staff in Chicago, Illinois.

Mr. Johnson, who is secretary of the Chicago Perfumery, Soap and Extract Association, was formerly purchasing agent for G. Barr & Co., Inc., manufacturing chemists of Chicago—a position he held for the past five years.

Increasing demand for its Lanolin, Wool Greases and Absorption Bases in many and varied fields of industry, has led N. I. Malmstrom & Company to expand research, production and sales facilities, in order to effect speedy handling of an expanding volume of business. Enlarged offices in Chicago, and an augmented sales staff for this region, assure prompt and efficient service to customers throughout mid-west and western territories.

James L. Johnson

RAPID METHODS OF GREASE ANALYSIS

(Continued from page 19)

the beaker well with petroleum ether. Rinse the beaker with 50 ml. of 10 per cent HCl and add it to the Erlenmeyer flask. A rubber stopper is inserted and the flask is shaken very thoroughly. The stopper is left in the flask so as to leave the contents under pressure. If the soap content is low, the solution may clear in 10 or 15 min., but with a grease containing 30 per cent soap it may take 1 or 2 hr. Let it stand, with occasional shaking, until the upper layer is perfectly clear and dissolved. Then transfer it to a separatory funnel and draw off the acid solution containing the Na, Ca, or K part of the soap. Wash the petroleum ether fraction at least six times, using 25 ml. of water each time to wash out all traces of acid. Usually by this time the wash water shows neutral when tested with blue litmus paper.

Next the petroleum ether fraction is transferred to an Erlenmeyer flask, the separatory funnel washed with petroleum ether, and washings added to the solution in the flask. At this point 25 ml. of neutral 95 per cent alcohol is added and the solution is titrated cold with 0.5 *N* alcohol potash using phenolphthalein as an indicator. Duplicate analyses should agree within 0.2 per cent.

Calculation of Soap Content

For example, for a 10-g. sample of grease, 9 ml. of 0.5 *N* alcoholic potash were needed. One cu. cm. of 0.5 *N* KOH = 0.028 g. KOH. Assuming, as A.S.T.M. does, that 1.0 g. of fatty acid requires 0.2 g. of absolute KOH for neutralization, the calculation would be made as follows:

$$9 \times 0.028 = 0.252 \text{ g. of KOH used}$$

$$\frac{0.252}{0.2} = 1.26 \text{ g. of fatty acid in 10 g. of grease or 12.6 per cent fatty acid}$$

If the soap were Na, to calculate to soap content:

$$12.6 \times \frac{305}{282} = 14.2 \text{ per cent Na soap}$$

If the soap were Ca:

$$12.6 \times \frac{604}{564} = 13.4 \text{ per cent calcium soap}$$

If the mixture of soaps were present, for very accurate work the ash would have to be analyzed and the soap content distributed in the proportions found there; but for ordinary determinations on ball-bearing greases a calculation based on Na soap should give results close enough, as it is doubtful whether the error in estimating the soap content would have any effect on predicting the performance of the grease in service.

Analysis of Grease (Sinclair Method)

Routine Method, as Applied to Driving Journal and Rod Cup Greases

By N. J. Gothard[1]

Under no circumstances is any part of the following method to be considered an alternative or substitute procedure for the A.S.T.M. Standard Method of Grease Analysis. The following procedures are intended for those routine control analyses wherein their degree of accuracy has been proved by experience to be sufficient. The following method is applicable only to straight sodium-soap greases containing no free fat, particularly to railroad types of grease of relatively high sodium-soap content.

[1] Sinclair Refining Co., East Chicago, Ind.

Petroleum Oil

A sample of 2.5 g. is weighed into a 125-ml. Erlenmeyer flask. About 30 ml. of 86 deg. naphtha is added to the flask, and the sample is broken up with a flat end stirring rod. The solution of oil in the naphtha is then decanted through an 11-cm. quantitative filter paper using slight suction and receiving the filtrate in a 500-ml. suction flask. To avoid possible loss of the naphtha solution, the stem of the filter funnel must extend into the suction flask well past the point of suction. The residue of soap in the Erlenmeyer flask is repeatedly broken up and washed with successive portions of naphtha until the soap is free of oil, whereupon the inside and outside surfaces of the Erlenmeyer flask are washed free of oil. Finally, the filter paper and inner surface of the filter funnel are washed free of oil with successive applications of naphtha. Care must be exercised that no oil is lost by "creeping" and that no flecks of soap pass into the filtrate.

The filter flask is then placed on a steam bath under a current of filtered air until the volume of naphtha solution is about 20 ml. With the aid of a naphtha wash bottle, the residue in the filter flask is completely transferred to a previously dried and weighed 150-ml. beaker. The best technique in this transfer consists in alternate washings of the outer surface near the mouth of the flask and the entire inner surface, avoiding any loss through the side arm of the flask.

After evaporation of the naphtha on a steam bath under an air current and drying for 1 hr. at 105 C. the beaker is cooled and weighed, and the per cent of petroleum oil calculated.

In a few cases, after experience has shown that consistently low results are obtained by using 86 deg. naphtha, due to the highly asphaltic nature of the oil, benzol may be substituted for the naphtha in the above procedure.

(Continued on following page)

RAPID METHODS OF GREASE ANALYSIS

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Free Alkali

A 2.5-g. sample is weighed into a 250-ml. Erlenmeyer flask. A second 250-ml. Erlenmeyer flask is used for the blank determination. Into each flask is placed a mixture of 100 ml. of 95 per cent alcohol (Formula No. 30) and 50 ml. of distilled water. There is then added to each flask exactly 10 ml. of 0.25 *N* hydrochloric acid, or an equivalent volume of HCl of other normality. The flasks are then stoppered with perforated corks carrying glass tube air condensers and placed upon a steam bath. The contents of the flasks are refluxed until the sample of grease has completely disintegrated.

The contents of each flask are then titrated with approximately 0.1 *N* NaOH or KOH solution, which has been standardized against benzoic acid. Phenolphthalein is used as indicator and the titrations are continued to a distinctly pink end point. If *A* is the ml. of blank titration, and *B* the ml. of sample titration, $(A - B) \times$ normality factor $\times$ 0.4

$$\frac{}{\text{Weight of sample}} =$$

per cent free alkali as NaOH

Total Alkali

A sample of 2.5 is taken and a determination of sulfated ash is made according to the procedure of Section 6, A.S.T.M. Standard Method D 128-37, entitled "Alternative Method for Ash." The following alterations of the published A.S.T.M. Method are made for this procedure:

(*a*) A platinum dish is substituted for a platinum crucible, and in no case is a porcelain crucible to be used.

(*b*) The ash is assumed to be sodium sulfate and is converted to NaOH for the purpose of percentage calculation as follows:

$$\frac{\text{Weight of sulfated ash} \times 0.5632 \times 100}{\text{Weight of sample}} =$$

total alkali as NaOH

Combined Alkali and Soap

Total alkali as NaOH—Free alkali as NaOH = combined alkali as NaOH

Combined alkali as NaOH $\times$ 7.625 = sodium soap, if beef tallow is source of soap.

Water

Use A.S.T.M. Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (D95-46).[2]

Calculation

(*a*) *Anhydrous Type.*—Determine free alkali, petroleum oil, and water. Deduct combined results from 100 per cent to obtain soap by difference.

(*b*) *Hydrous Type.* Determine soap (as in section on Combined Alkali and Soap), petroleum oil, and free alkali. Deduct combined results from 100 per cent to obtain water and glycerin by difference.

Determination of Sodium Soap in Greases Known to Contain Only Sodium Soap and No Interfering Fillers

By W. S. Palmer[1]

The rapid control method for the determination of sodium soap in greases known to contain only sodium soap and no interfering fillers shall consist of igniting a suitable quantity (2 to 5 g.) of the grease in a platinum or porcelain crucible to a dry carbon. This can best be done by means of a Bunsen burner held in the hand and the sides of the crucible heated until the grease has melted and ignited. Continue to heat the grease gently, supplying only enough heat to make it continue burning, until all the volatile matter has burned away and the dish and contents are at a dull red heat. Remove the crucible and contents at once when this stage has been reached, place in a pyrex beaker filled with distilled water, cover with a watch glass and boil for 2 hr. on a hot plate, disintegrating the carbonaceous residue completely with a stirring rod. At the end of this time determine the total amount of alkali present by titrating the cooled solution with 0.25 *N* hydrochloric or sulfuric acid, using methyl orange as an indicator. From the results of this titration the sodium soap may be calculated by use of a suitable factor found by experience to fit the particular series of products under test. Of course, the amount of free alkali present in the sample will have to be taken into account and corrections made for it. For quick control analysis, this system of anaylsis obviates the necessity for separating and testing the combined fatty acids.

[1] The Texas Company, Port Arthur, Tex.
[2] 1946 Book of A.S.T.M. Standards, Part III-A, p. 331.

BARIUM LUBRICATING GREASE

(Continued from page 2)

on the other hand do hydrol[illegible]g in wate[illegible] so that you may have both free base and free fatty acids. Some calcium soaps have this tendency also.

The qualities of Barium Lubricating Grease which make it a versatile lubricant are both water and heat resistance, sufficient cohesion so that it does not drip, ability to maintain consistency on bearings, sufficient adhesion so that it resists centrifugal action, soft enough consistency so that it can be pumped with ordinary power-operated grease guns at temperatures as low as 35° F. and by hand guns at a lower temperature, and lastly ability to prevent rust. These properties make this a lubricating grease with wider applications than others now available. Eastern steel mills have records of numerous applications of this lubricant, which indicate it is practically an all-purpose lubricating grease. Mining companies, smelters, cement plants, gravel plants, rock crushing plants, packers, canners, breweries and construction companies standardized on this lubricant. As an indication of the versatile nature automotively of this grease, we have assigned the name "4-*in*-1" Barium Grease to this product. This indicates that in automotive applications one lubricating grease can be used for Chassis . . . Wheel Bearings . . . Water Pump . . . and Universal Joints lubrication. It also is an excellent covered spring lubricant. The advantages of an all-purpose lubricant are quite evident, for with one drum of grease instead of several, less space is occupied and greater cleanliness is possible. Further, there is little chance of using the wrong type of grease. Service stations have found an economy in using this lubricant in that about two-thirds as much "4-in-1" Barium is required as of all other greases to lubricate a given

number of cars. To date, several million pounds of this lubricant have been used for automotive lubrication with outstanding success.

Application of Barium Grease

You will be interested in some specific applications in your own and other industries. An eastern steel mill by replacing several other greases with Barium Lubricating Grease finds less frequent greasing necessary, a lower grease consumption, and less error in greasing. Specifically: On their 44 in. bloomer screws, when rolling slab steel, it was necessary to shut the mills down several times a day to cool the screws. They now make mixture of "4-in-1" Barium Grease with a small amount of steam cylinder oil to increase the fluidity of the lubricant. Using this, they can now operate a full shift without shut-down due to the fact that the Barium Lubricating grease keeps the screws lubricated under extreme working conditions and high temperatures; on their 54 in. bloomer pit covers, where formerly it was necessary to lubricate the wheel bearings on the travel mechanism once or twice a day, since changing to Barium it is only necessary to lubricate once a week with added ease of movement; Barium is also used on ingot transfer buggy journals moving slab from soaking pits to bloomer entry tables. Formerly, it was necessary to lubricate these journals several times a day and then difficulty was encountered in moving the cars causing loss of time and damaged bearings. Since using Barium, it is only necessary to lubricate two or three times a week, and there is no difficulty in moving cars; on the core and ingot mold oven cars in the foundry there was formerly much damage to cores and molds due to the fact that bearings would freeze and in an attempt to move the cars, the molds or cores would be broken. Average temperature in these ovens is about 500° F. the cars being at this temperature for about fourteen hours. The cars are now lubricated with Barium before being placed in the ovens and in no case after such lubrication have any of the bearings frozen and all core and mold breakage due to rough handling of cars has been eliminated. On cold mill annealing furnace fans operate under the furnace in conducted temperatures of approximately 1600° F. It was formerly necessary to lubricate fan bearings many times a day. Since changing to Barium, it is only necessary to lubricate the fan bearings once a day; and, in most cases, once every two days. Since the use of this latter lubricating grease was started, there has been no loss of bearings. On up-drafts fans over the boilers of this plant the bearing temperatures will run approximately 400° F. on the high-speed turbine driven fans (turbine end 5000 rpm, fan 750 rpm). It was formerly necessary to climb to the top of the boilers and lubricate daily. Since using Barium, the bearings are lubricated once a week and there has been no bearing loss during the past year. In a western steel mill we have advice that the use of Barium Lubricating Grease has simplified the problem of lubrication. It is also being used on several miles of conveying system, coke and skip cars, pusher machines, mud guns, door machines, shaker screens, etc.

Barium Lubricating Grease has been successfully used on flat steel and boiler plate in supply yards as well as on steel parts of towers, bridges, etc. for protection against moist air, salt water spray and even the direct impact of ocean waves. It has also successfully lubricated the governor control vanes regulating water flow to turbines where it displaced calcium base grease, which washed off with consequent corrosion. Dredging companies have also found that Barium gave better service than other lubricants. The heavy trucks of a strip coal mine often have to travel through water and due to the loads carried and solvent action of the water, wheel bearing trouble was encountered until Barium was employed. One of the most interesting ap-

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BARIUM LUBRICATING GREASE

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plications of Barium Base Grease was on the conveyor system used for moving aggregate at the Mt. Shasta Dam. On the main conveyor and plant lines there were approximately 85,000 bearings. Much of the equipment was exposed to atmospheric temperatures ranging from 10 to 130° F. throughout the year, and the rainfall at times during the winter months was very heavy. In addition, dust and sand was always present in the vicinity of the bearings. In spite of such conditions this equipment operated most successfully for 3½ years.

In the plants of the packing or canning companies, the versatility of Barium Grease has been utilized. It is necessary in these plants to clean the equipment thoroughly at regular intervals during each operating day with water, steam and detergent. Many of the bearings operate in a wet or humid atmosphere at temperatures up to 200° F. Under these conditions, it is essential that the lubricant used in the bearings be both water and heat-resistant and protect the bearing surfaces against corrosion and that it should not be displaced from the bearing during the regular washing down operation with steam, water, and detergent. Furthermore, in some of the plants the severely corrosive action of certain fruit juices is a matter of major concern, and any grease that offers protection against corrosion is particularly valuable. Under all of the above conditions, Barium Grease has been used with highly gratifying results.

Of course, mining companies have taken advantage of this versatile lubricant and one illustration of the use at a copper smelter will be of interest. The problem involved the lubrication of bearings on a conveyor moving blister copper from the furnace to the cooler. Because of the heat from the copper slabs, the steam and moisture attendant to the dousing of the slab for cooling, and the operation of a section of the conveyor under water at 200° F., satisfactory lubrication of the equipment presented a difficult problem. Thirty-two lubricating greases were tested and Barium was the only one found satisfactory for the purpose. Lubrication costs were cut to one-quarter and the frequency of lubrication reduced from twice daily to once every four days.

We do not claim that you can always obtain such outstanding results, but we do feel that you can lick many of your stubborn lubrication problems with Barium Lubricating Grease.

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